I. RADULESCU, A. V. RADULESCU, F. VASILIU

RESEARCH

The Structure of Lubricating Greases by Electron Microscopy

The paper presents experimental researches concerning the microscopical structure of the lubricating greases, for different chemical composition. The investigations given in this paper have potential to diagnose the behavior of the greases in various dynamical conditions. The results are compared with those published in the literature.

Key words: *lubricating greases, structure, electron microscopy*

1. INTRODUCTION

Each substance molecula has its own radiation length of wave and their analyse may establish the product quantity.

The electronic microscopy method for the fresh lubricants structure determination was used from 1941, by Mc. Bain, to find improvements for the fabrication technology, [3]. The special interest established by the microscopic studies results, the improved devices and methods leads to series of studies concerning the greasses structure in different exploitation stages.

Most automotive and industrial lubricants contain additives designed to enhance total product performance. These added materials are present in many forms, from the all-pervading soap network of a grease, through the high-molecular-weight polymers used to improve the viscosity index, to the nanometre sized detergent micelles present in an engine oil. During use, additional material, in the form of soot, additive degradation products and wear debris, may also become incorporated. Although optical micoscopy may be sufficient to observe some of the larger agglomerations of particulate material arising from extensive lubricant use, the greater resolving power of the electron microscopy is required to image the majority of particles, especially during the early stages of degradation.

Eng. Irina RADULESCU,
S. C. ICTCM S. A. Bucharest, Romania
Dr. Eng. ALEXANDRU V. RADULESCU,
University „POLITEHNICA” Bucharest, Romania
Dr. Phys. Florin VASILIU,
INCDFM Bucharest – Magurele, Romania

2. TECHNIQUES AND RESULTS

The specific process is the extraction of insolubles in a form suitable for introduction into the vacuum environment of an electron microscope and it is not very difficult, but it has been necessary therefore to develop a variety of complementary techniques to effect complete characterization of insolubles in lubricants. That’s why there is a number of techniques which have proven to be of value in the preparation of specimens containing mineral oil and greasses, for examination in scanning (SEM) and transmission (TEM) electron microscopes, [4], [7].

Using TEM the enlargement is variable between 8000 X-200.000 X, function the product nature and the investigation purpose.

The electronical scanning microscopy is a simple electrono-microscopic technique, with advantages and also disadvantages concerning the electronic microscopy and it is complementary to it (Table 1).

The image realised in these apparatus is made using a fine and tight spot of electrons which „sweeps” the studied surface.

The X radiation emission is given by the interaction between the electronic spot and the solid body – it is used in X radiation spectral microanalysis.

Emited X radiations are given from a volume about few μm^3 near the surface. It is possible to obtain the qualitative analyse of demanded tests because each element has it’s own X radiation lines set and it can be identified it’s own wave length or it’s element characteristic lines energy.

Tabel 1. Working manner in scanning electronic microscopy

Working manner	Gathering signal type	Supply contrast type and information	Spacial Resolu-tion
Reflexion	Reflected electrons	Compositional Crystallographic	100 nm
Emision	Secundary electrons	Topographic Potential Electric and magnetic fields	10 nm 100 nm 1 µm
Lightning	Photons	Compositional	100 nm
Conduction	Testing electric power	Conductibilitate indusă	100 nm
Absorbtion	Testing absorbed electric power	Topographic	1 µm
X Radiation	X photons	Compositional	1 µm
Auger	Electrons	Compositional	1 µm
Transmi-ssion	Electrons transmi-ssion	Crystallographic	1– 10 nm

Also, focusing the electronic spot into a certain point, it is possible to realize a qualitative analyse indicating present chemical elements. This allows to establish the different elements distribution limited by a certain investigation surface.

The quantitative analyse consists in present elements concentration determination into a test analised minivolume. This is possible using specialised meters or similar analogical devices for a direct X radiation intensity evaluation.

This method is using special apparatus „electronical sounding lead" and the analyse is made for a small substance volume, function of electronical spot diameter, medium atomic number and acceleration tenssion. This is defined for a minimum detectable of substance: 10^{-15} – 10^{-16} g, and quantity evaluation precission, after corrections is about $\pm$ 1%.

3. ELECTRONIC MICROSCOPY EXAMINATION TECHNIQUES

Lubricating grease is defined by the American Society for Testing and Materials (ASTM) as a solid or semi-fluid lubricant consisting of a thickening agent in a liquid lubricant. It is important to improve certain properties and functions, and also, the additional components sometimes included (the additives), [1].

Lubricating grease properties depend on both its composition and the manufacturing process used. Using SEM - Scanning Electron Microscopy it is possible to obtain structural network images of the metallic soap into the grease. The prepared tests methods consists in, [7]:

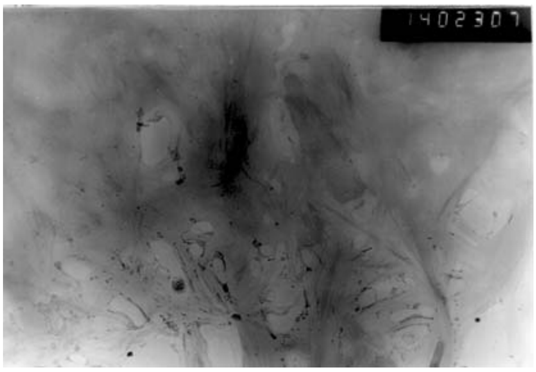

Figure 1: L95 Ca3/4 G grease

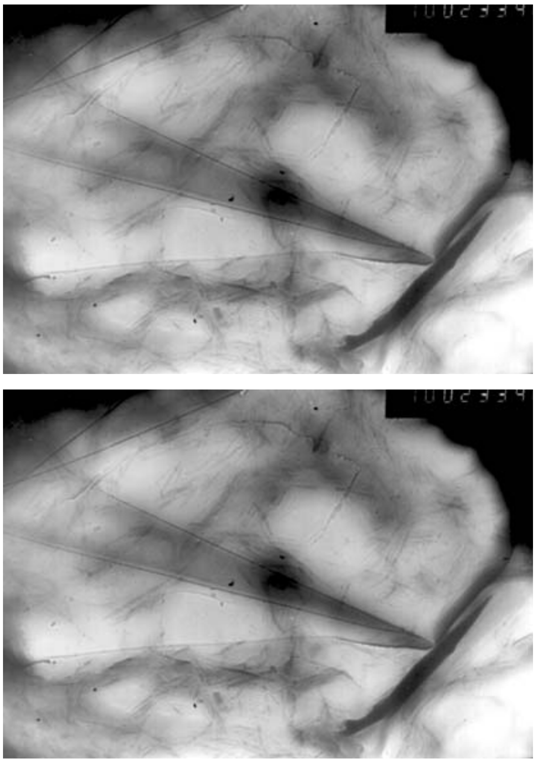

Figure 2: U85 Ca3 grease

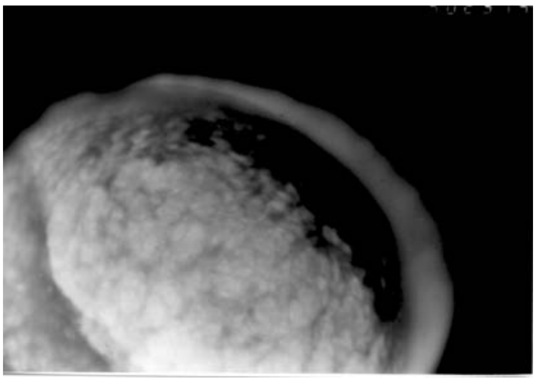

Figure 3: UM 175 LiCa 3 grease

- mineral oil detachment by disolving in heptan and obtaining a tridimensional soap matrix; (Fig. 1 - L95 Ca3/4 G grease and Fig. 2 – U85 Ca3 grease);
- mineral oil detachment by filter paper absorbtion action; (Figure 3 - UM 175 LiCa 3 grease)

Figure 4: UM 185 Li0 grease

- mineral oil freezing from the grease structure, at the temperature -100 ^{0}C; (Figure 4 - UM 185 Li0 grease)

The greases quality and performances are essencially influenced by the dimensional and spatial soap fibers distribution. Using electronical microscopy it is necessary to examine the additives and the soaps, which concure to the high performance quality greases, [2].

Lubricating greases consists in a soap as a solid or semifluid lubricant agent, into a liquid lubricant. The microstructure of the soap phase has the shape of fibres, which imobilise great quantity of liquid into their framework.

The mineral oil into the soap fibres network is like a jelly which become deformed applying cutting forces and come back to the solid statement after the forces dissapear.

Soap fibres are 80 nm thickness and consists in Ca and Li salt of hydrostearic acid, the greasses quality and performances are influenced by their dimensional and spatial distribution.

On the other hand, the solid insoluble elements majority is too small to visualize using optical mycroscop, being necessary the electronic microscope and its high resolution - for the morphological examination of the additives and thickening elements needed in the quality greasses performance.

4. ELECTRONIC MICROSCOPY STUDIES CONCERNING LUBRICATING GREASSES

Mineral oil is one of the most intractable contaminants of the microscope vacuum, as the pumps are unable to deal effectively with it. Decomposition of this oil vapour in the electron beam results in carbon being deposited onto the specimen. In order to examine the insolubles in specimens containing mineral oil, the oil must be removed or its vapour pressure reduced to a negligible value.

A fine 3 mm-diameter copper grid is used to support the specimens in the TEM and this grid, when covered with a very thin (0.04 fJ.m) film of carbon, retains small particles, particularly soot.

The carbon film grid is simply dipped into the mineral oil and then into a solvent (eg. heptane). Clearly, particles which do not have an affinity for the carbon film will be, to a large extent, lost. On the other other hand, the smaller(< 1 μm) particles, especially those of soot, adhere extremely well and can be examined at fairly high resolution (Figure 5).

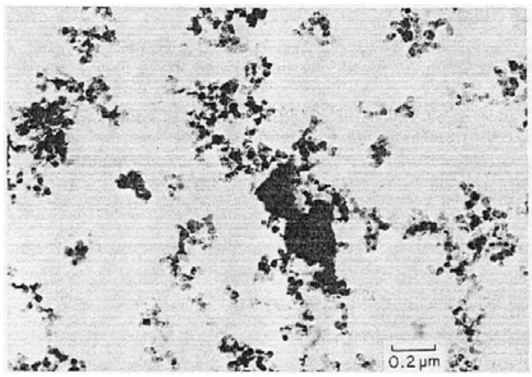

Figure 5: Soot particles adhering to a carbon film, [7]

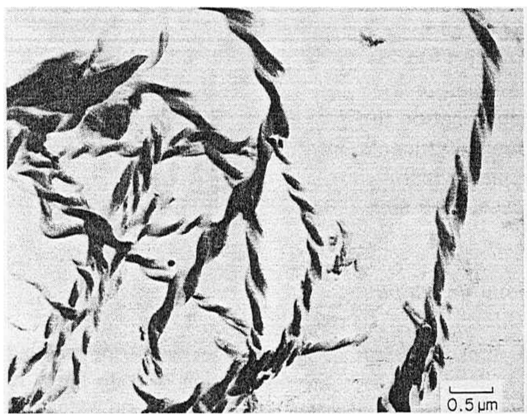

Figure 6: Platinum-shadowed grease-soap fibre, [7]

The deposit (in the case of Figure 6, these are grease-soap fibres) can be shadowed with evaporated platinum to gain height information. However, because this method is primarily suitable for the collection of small particles, it does not provide a representative sample of the particle population and other methods are required to give a fuller picture.
The solvents used were acetone (not succesfull) and extraction gas, but better results it may be obtain using heptan.

5. TRANSMISSION ELECTRONIC MICROSCOPY EXPERIMENTAL RESULTS –CONCERNING GREASSES THERMICAL DEGRADATION

Researches concerning greasses thermical degradation had objectifs the study of several greasses types, the experimental results were obtained using TEM, [5], [6].

It is possible to visualize, by this method, the soap fibres network and other insoluble solid elements, but the tests must be suitable prepared. Best results were obtained using extraction gas and heptan – as solvents.

Analysing following greasses (figures 7 ...12) it is obvious that the fibres net is less homogeneous in Figure 7 – U 85 Ca 3 and more homogeneous in Figure 8 – U 90 Ca 1, the fibres thickness is around 50 – 100 nm. Both cases there were some areas where the soap fibres tridimensional network was not corresponding to an adequate quality greasse.

Nevertheless, the comparative analyse indicate an advantage for greasse U 90 Ca 1 - which has a more dense soap fibres net distribution; that’s why it is assured the greasse cutting stability during lubricating.

Optimum fibres shape is induced by the soap composition, the peculiar used oil, processing way.

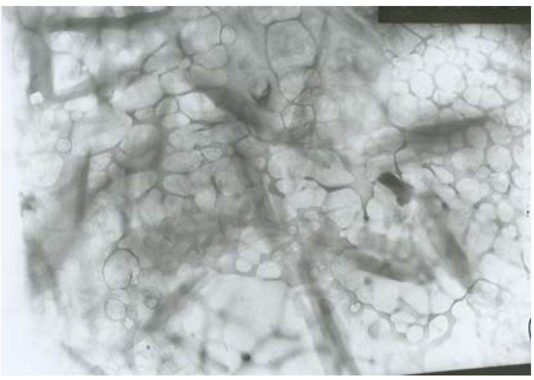

Figure 7: U85 Ca 3 grease

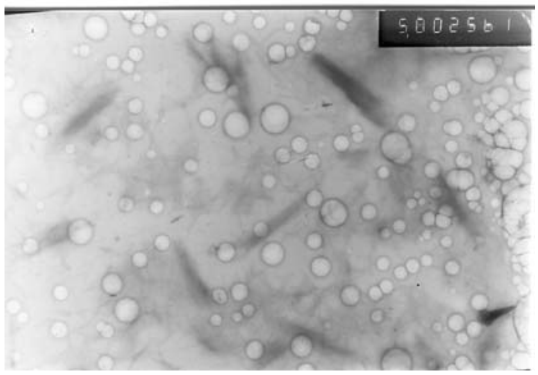

Figure 8: U 90 Ca 1 grease

It is difficult to prepare the electronic microscopy greasses tests. Best results were obtained using the method with heptan or hexan as solvent. The aspect of the fibre structure is identical with the one from speciality literature [1].

Advanced and coherents mineral fibres networks, with high conectivity degree is cleary seen in Figures 9 and 12, which make a similarity to a good rheological behaviour of those greasses.

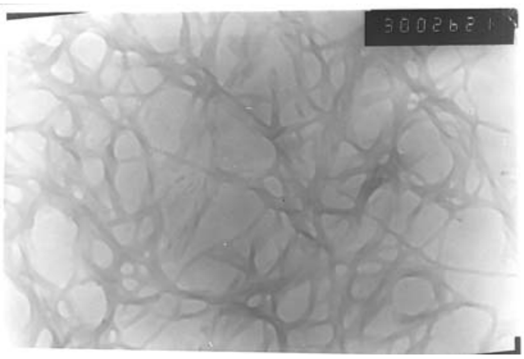

Figure 9: U 185 L iEP grease

The tests from Figures 10 and 11 have a peculiar structure, with some fibres chains, which consist in small extension networks.

Their homogenity is low, and, if there are some fibres, those are too short to realize a net – or it is made an open net, with coherence and conectivity in a small degree.

The absence of mineral fibres reticular structures into studied greasses may notice a different structure or an adequate TEM tests preparation, in difficult cases.

The results obtained using the electronic mycroscop and studying the datas from speciality literature guides to the conclusion concerning the best rheological behaviour to the greasses from Figures 9 and 12 and a weak behaviour expected to tests from Figures 10 and 11.

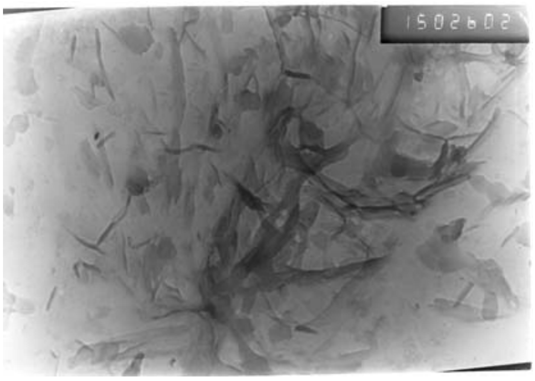

Figure 10: RUL 145 grease

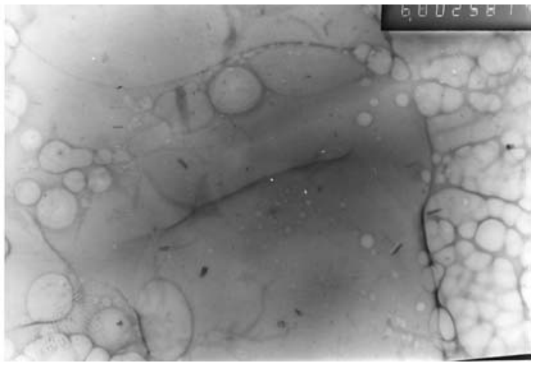

Figure 11: RUL 165 Na 3 grease

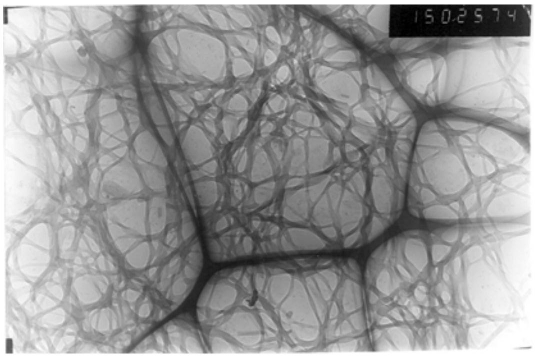

Figure 12: Shell R2 grease

6. CONCLUSIONS

1. Microscopical structure of lubricating greases data base is not only a software project, but it is also a material, informational, methodological and knwoledge resourse system, useful in scientifical reaserch developing.
2. It can be developed using the international experience and it follows a better contact between the roumanian research and education and the international values circuit.
3. The results of these investigations will be an aide in the fundamental or application research and also they will assure the documentation at high level instruction and consulting for different beneficiaries.
4. The viability and the success of this data base are guaranteed by the novelty of the idea - to diagnose the behaviour of several tested lubricating greases.
5. The achievement of the project is the informations data base concerning the behaviour of those greases, in various dynamical conditions and the possibility to enrich it every moment.

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